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High-power and long-life lithium-ion batteries using lithium titanium oxide anode for automotive and stationary power applications

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HIGHLIGHTS

► LTO system cells have been developed for automotive and stationary power applications.
► 3 Ah-class LTO/LMO cell designed for 64 Wh kg^{-1} exhibited a high-power density of 3600 W kg^{-1}
► Long-life performance of the LTO/LMO cell is suitable for HEV and ISS applications.
► 20 Ah-class LTO/NCM cell designed for 90 Wh kg^{-1} exhibited a long storage performance.
► The LTO/NCM cell is suitable for not only EV but also PHEV and stationary power applications.

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ABSTRACT

Lithium-ion batteries with a combination of a lithium titanium oxide (LTO, $Li_{4/3}Ti_{5/3}O_4$) anode and 4-volt-class cathodes, namely, $LiMn_2O_4$ (LMO) and $LiNi_xCo_yMn_{1-x-y}O_2$ (NCM) cathode, have been developed for automotive and stationary power applications. The 3 Ah-class LTO/LMO cell for high-power applications had a high output power density of 3600 W kg^{-1} for 10 s pulse. High-rate full cycling tests at 10 C rate exhibited a high capacity retention of 95% after 30000 cycles. The capacity retention after 15 years at 35 °C in the calendar life tests is predicted to be 93%. Such a high-power and long-life performance of LTO/LMO cell is suitable for not only hybrid electric vehicle (HEV) but also idling-stop system (ISS) applications. The 20 Ah-class LTO/NCM cell with an energy density of 90 Wh kg^{-1} exhibited a power density of 2200 W kg^{-1}. The storage life for floating charge at 100% state of charge (SOC) and the full cycle-life at 3 C rate are predicted to be 10 years at 45 °C and 10,000 cycles, respectively. It was demonstrated that the LTO/NCM cells have an excellent balance of high-power, high-energy, low-temperature, and long-life performance for not only electric vehicle (EV) but also plug-in hybrid electric vehicle (PHEV) and stationary power applications.

1. Introduction

Lithium-ion battery systems with a combination of the LTO ($Li_{4/3}Ti_{5/3}O_4$) anode and cathodes such as $LiCoO_2$ (LCO) [1–3], $LiNi_xCo_yMn_{1-x-y}O_2$ (NCM) [3,4], $LiMn_2O_4$ (LMO) [5–9], and $LiFePO_4$ (LFP) [10,11] have been proposed as they offer attractive cell chemistry and are expected to realize high-power, long-life, quick-charge, and safety for large-format battery applications such as ISS, HEV, PHEV, EV, and stationary power systems because the LTO electrode has advantages, namely, a zero-strain property, nano-size particle, no lithium plating at quick charging, and thermal stability in high-temperature conditions. In particular, limitations on high-power, capacity, and life of lithium-ion batteries are important in the context of applications to vehicles and stationary power systems. Long-term cycling, high-temperature storage, and quick charging accelerate large degradation of the output power and the capacity of conventional lithium-ion battery systems using graphite anode. LTO/LMO and LTO/LFP cells have recently been proposed as promising batteries with high-power and long-life for HEV and stationary power applications.

We have studied the electrochemical kinetics concerning lithium insertion and extraction for LTO particles [3,13] in order to understand high-rate performance and also reported the performance and safety of prototype LTO cells using 4-volt-class cathodes of LCO [3,12], LMO [6,7], and NCM [3]. Although the discharge rate

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capability of the NCM cathode is inferior to that of the LMO cathode, the NCM cathode has an advantage in that its capacity can be larger than that of the LMO or the LCO cathode. Therefore, we have been developing two type of LTO/LMO cell for high-power and LTO/NCM cell for high-energy applications. This paper describes the cell performance of the LTO/LMO and the LTO/NCM system for automotive and stationary power applications.

2. Experimental

Prototype 3 Ah-class LTO/LMO laminated cell was constructed by using the LTO anode, the cathode of LMO-based material, an organic electrolyte, polyethylene separator, and a laminated film case. Prototype 20 Ah-class LTO/NCM prismatic cell using an aluminum can case was also constructed by using the LTO anode, the cathode of NCM-based material, the organic electrolyte, and the polyethylene separator. The LTO power was a $Li_{4/3}Ti_{5/3}O_4$ spinel sample with an average diameter of about 0.9 μm obtained by a conventional solid–state reaction method. The LMO-based material and the NCM-based material mostly contained $LiMn_2O_4$ and $LiNi_{0.5}Co_{0.2}Mn_{0.3}O_2$ from a commercial sample, respectively. The LTO anode, the cathode construction, and the laminated film case were the same as those described previously [3]. The LTO anode was prepared from a past of LTO, carbon, and polyvinylidene fluoride binder mixture in n-methyl pyrrolidone. The past was coated on an aluminum foil (15 μm thickness) as the current collector. The LTO anode contained 6 wt% carbon and 2 wt% binder. The density of LTO anode was 2.2 g cm^{-3}. The anode–cathode capacity balance was set to be equal. The organic electrolyte consisted of a mixture of propylene carbonate (PC) and diethyl carbonate (DEC) solvent containing lithium hexafluorophosphate ($LiPF_6$).

The rated capacity of the LTO/LMO cell was measured at 25 °C by a discharge current of 3 A down to 1.5 V after CC–CV (constant current–constant voltage) charging at 3 A constant current up to 2.8 V of constant voltage to 0.15 A of cut-off current. The minimum rated capacity of the LTO/LMO cell was 3 Ah for the production variability. In the cell performance tests, we selected that 1C rate is 3 A. The rated capacities of the LTO/NCM cell were also measured at 25 °C by the discharge current of 20 A down to 1.5 V after CC–CV charging at 20 A up to 2.7 V to 1 A, respectively. The DC resistance and the power densities of output and input for 1, 10, and 30 s were calculated from the results of the Hybrid Pulse Power Characterization (HPPC) test [14]. The maximum pulse current selected was a 5 C rate on the basis of the low current HPPC test. Galvanostatic charge pulse tests of LTO (26 μm thickness) and graphite anode (20 μm thickness) at 80% SOC and −30 °C were performed with a three-electrode glass cell constructed using the anodes (2 × 2 cm, 3.4 mAh capacity), a Li metal chip reference electrode, and the LCO cathode. The electrolyte selected in the graphite anode cell was a mixture of ethylene carbonate (EC) and DEC solvent (1:2 by volume) containing $LiPF_6$, which does not freeze at −30 °C.

Calendar life tests of LTO/LMO cells stored at 35 and 45 °C after charging to 2.8 V were carried out by measuring the recovery capacity and the DC resistance for 10 s every week. High-temperature storage-life tests of LTO/LMO cell and graphite/LMO cell stored at 60 °C after charging to 100% SOC were carried out by measuring the recovery capacity every week. Self-discharge tests of the LTO/NCM cells stored at 25 and 45 °C after charging to 2.7 V were carried out by measuring the residual capacity and the recovery capacity after storage for 19, 39, 67, 188, and 365 days. Floating-charge storage tests of the LTO/NCM cells during charging at a constant voltage of 2.7 V and at 35 and 45 °C were carried out by measuring the residual capacity and the DC resistance every two weeks.

Charge–discharge cycling tests of the LTO/LMO cell and the LTO/NCM cell were carried out between 2.8 and 1.8 V at 10 C (30 A) rate and between 2.7 and 1.5 V at 3 C (60 A) rate at 25 °C, respectively.

3. Results and discussion

3.1. Performance of 3 Ah-class LTO/LMO cell for high-power use

3 Ah-class LTO/LMO cells have been developed for high-power use such as HEV and ISS applications. The specifications of the LTO/LMO laminated cell are summarized in Table 1. The nominal capacity, the nominal voltage, and the energy density were 3.3 Ah, 2.5 V, and 64 Wh kg^{-1}, respectively. Typical discharge curves of LTO/LMO cell at various discharge rates from 1 to 50 C are shown in Fig. 1. The discharge curve at 50C rate showed high capacity retention of 94% and flat voltage. Such a high-rate capability is attributed to three-dimensional lithium diffusion from the spinel structure of small LTO and LMO particle. Fig. 2 shows discharge performance at various temperatures from −40–45 °C. The discharge rate was 1 C rate. Even at −40 °C, the discharge capacity maintained 80%. Excellent low-temperature performance at −40 °C is suitable for automotive applications. PC-based electrolytes without ethylene carbonate (EC) solvent are used for LTO cells, which is advantageous for operation in low-temperature conditions compared with lithium-ion cells using graphite anode and EC-based electrolytes since EC solvent freezes.

Power capability and life performance of LTO/LMO cell were evaluated for HEV and ISS applications. Fig. 3 shows pulse-power capability for LTO/LMO cell obtained by HPPC testing at 25 °C. The maximum output power density at 50% SOC for 1 s and 10 s were 6600 and 3600 W kg^{-1}, respectively. The maximum input power density at 50% SOC for 1 s and 10 s were 6200 and 3500 W kg^{-1}, respectively. The maximum pulse-power density at 50% SOC of more than 3000 W kg^{-1} in the short-time range from 1 to 10 s is high enough to be competitive with conventional HEV in terms of driving performance. The power density for 10 s of more than 2600 W kg^{-1} was maintained in the wide SOC range of 10–90%. Such a flat pulse-power performance will allow a large available capacity and small battery size compared with high-power lithium-ion cells using hard carbon anodes for HEV applications [15,16]. The flat pulse-power performance is attributed to the maintaining of the flat open-circuit potential of LTO anode and LMO cathode in the wide SOC range.

Fig. 4 shows cycle life performance of LTO/LMO cell by high-rate charge–discharge cycling at 10 C (30 A) rate between 1.8 and 2.8 V in 100% SOC width. At atmosphere temperature of 25 °C, the maximum temperature at the cell surface during cycling increased from 32 to 37 °C. The high-rate cycle life test exhibited the long cycle life performance with the capacity retention of 95% after 30000 cycles. Even at high-rate charge–discharge operation for HEV and ISS use, the LTO/LMO cell showed only slight capacity fading because there is no lithium metal plating on the LTO anode.

Table 1
Specifications of 3 Ah-class LTO/LMO cell for high-power applications.

Cell	3 Ah-class LTO/LMO cell
Case	Laminated film
Nominal capacity at 1 C (Ah)	3.3
Nominal voltage (V)	2.5
Dimensions (mm)	120 × 72 × 8.5
Weight (g)	128
10s output power (W kg^{-1})[a]	3600
10s input power (W kg^{-1})[a]	3500
Energy density (Wh kg^{-1})	64

[a] At 50% SOC and 25 °C.

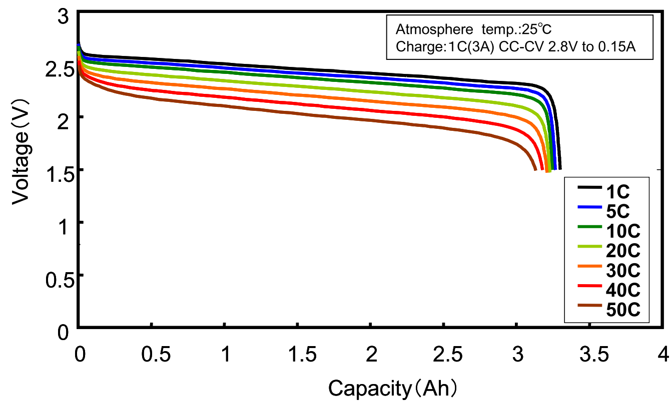

Fig. 1. Discharge curves of the LTO/LMO cell at various C rates.

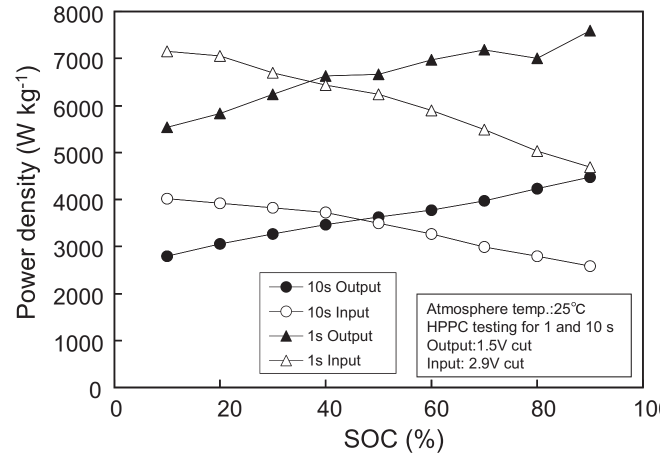

Fig. 3. Power capability of the LTO/LMO cell for 1 and 10 s at 25 °C by HPPC testing.

The capacities as a function of the square root of cycle number and cycle time are plotted in Fig. 5. The capacity fading had a linear relationship with the square root of cycle number and cycle time after 10,000 cycles. Cycle number and cycle time at the capacity fading of 10% in accordance with the square root of cycle number and time rule are predicted to be 200,000 cycles and 15 years, respectively. Such a long cycle life performance indicates that the LTO/LMO cell has good durability against not only a high-rate pulse cycling in HEV use but also a full discharge-charge cycling in ISS use.

Fig. 6 shows calendar-life performance of LTO/LMO cells stored for 600 days at 80% SOC. The capacity retention and the DC resistance ratio after 600 days and 35 °C were 97% and 1.05, respectively. The capacity fading and the DC resistance rise of LTO/LMO cells at 35 and 45 °C also had a linear relationship with the square root of time. The capacity retention and the DC resistance ratio after 15 years at 35 °C are predicted to be 93% and 1.2, respectively. If the end of life (EOL) for HEV and ISS use is time to reach 90% capacity retention, the LTO/LMO cell is expected to have the 15-year calendar life at 35 °C. It is noted that the capacity fading during the cycling test at 32–37 °C in Fig. 5(b) on the basis of the relationship with the square root of time is similar to that during the calendar life test in the atmosphere at 35 °C in Fig. 6. These results indicate that the capacity fading for the LTO/LMO cell during the storage and cycling tests is mainly dependent on not the stress of charge–discharge cycling tests but the calendar life. We consider that irreversible reactions such as lithium metal plating and film formation on the LTO anode during any cycling tests with low-temperature, high-rate charge, or wide SOC range operation are relatively negligible compared to those on the cathode, and then the film formation reaction on the LMO cathode mainly leads to the capacity fading. Therefore, the EOL for the LTO/LMO cells in the operation can be simply evaluated by characterization of the calendar-life for the LMO cathodes. It is well known that LMO cathode in lithium-ion cells is subject to the problem of Mn dissolution and deposition on the graphite anode that causes a large capacity fading in high-temperature storage conditions. Fig. 7 shows high-temperature storage performance of LTO/LMO cell and graphite/LMO cell at 100% SOC and 60 °C. The graphite/LMO cell had large capacity fading caused by Mn dissolution in 6 weeks. The LTO/LMO cell showed good high-temperature storage performance with 90% capacity retention after 20 weeks. We consider that no electrochemical Mn metal deposition on LTO anode at 1.55 V vs. Li suppresses the capacity fading of anode and cathode in LTO/LMO cell in high-temperature conditions.

For HEV and ISS use in low-temperature conditions, it is necessary to prevent lithium metal from plating on the anode during input power operation or quick charging. Fig. 8(a) shows potential profiles of LTO and graphite anode in three-electrode cell during 10 C rate pulse charge at 80% SOC and −30 °C and the minimum potentials of anodes at various pulse rates. The graphite anode at 10 C rate pulse charge in Fig. 8(a) shows much lower potential profile vs. lithium metal potential, indicating lithium metal plating on the graphite anode. The LTO anode shows much higher potential profile, indicating no lithium plating. Even at 25 C

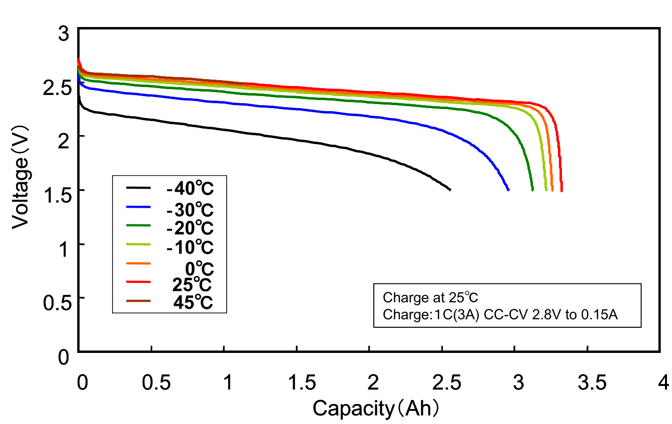

Fig. 2. Discharge curves of the LTO/LMO cell at various temperatures.

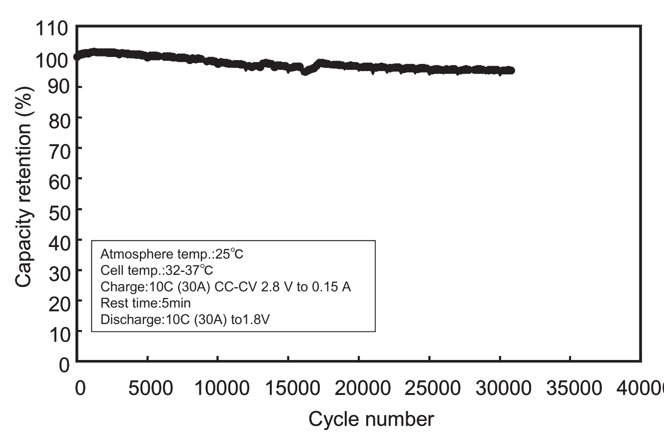

Fig. 4. Capacity retention as a function of cycle number for the LTO/LMO cell at 10 C rate charge–discharge cycling between 1.8 and 2.8 V in 100% SOC width.

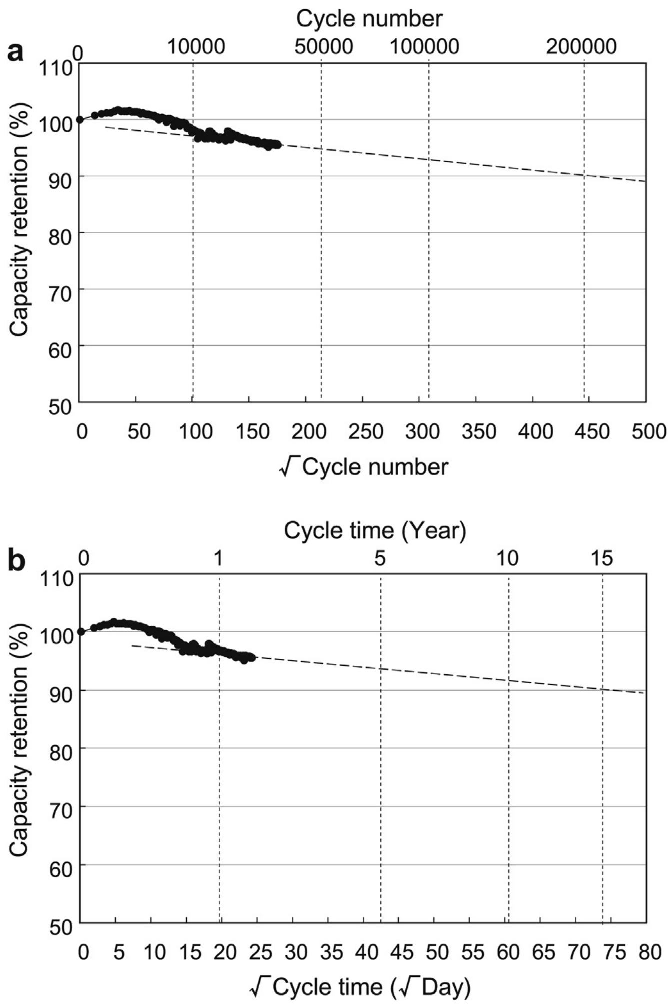

Fig. 5. Capacity retention as a function of √cycle number (a) and √cycle time (b) for the LTO/LMO cell obtained from the cycle life test in Fig. 4.

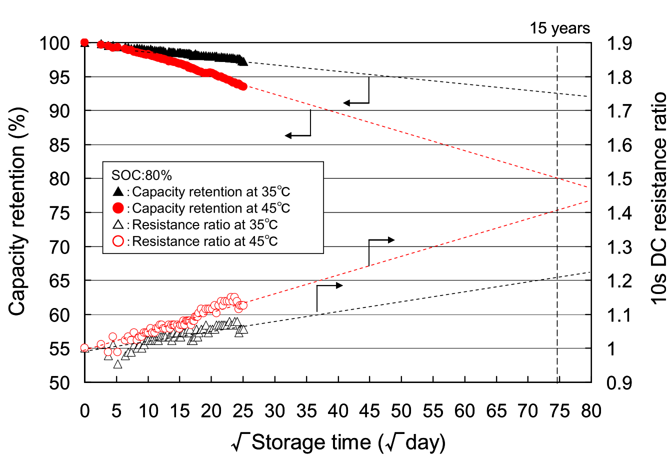

Fig. 6. Capacity retention and DC resistance ratio as a function of √time for the LTO/LMO cells obtained from the calendar life test at 80% SOC.

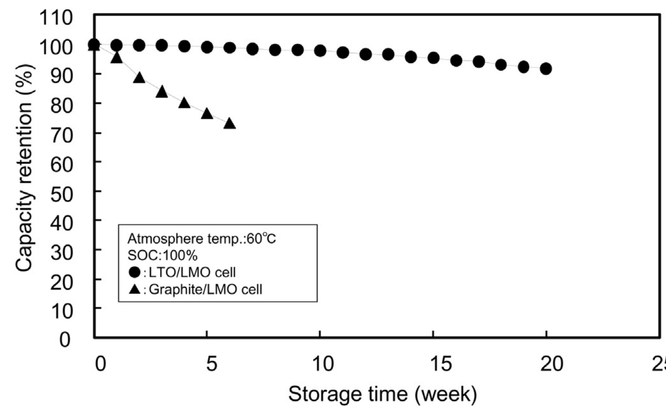

Fig. 7. Capacity retention as a function of storage time for the LTO/LMO and the graphite/LMO cell at 100% SOC and 60 °C.

rate, the minimum potential of the LTO anode was higher than lithium metal potential as shown in Fig. 8(b). Therefore, LTO cells have an advantage for use up to the high SOC range for HEV and ISS applications in low-temperature conditions because there is no degradation and damage of cell caused by lithium metal plating on the anode.

3.2. Performance of 20 Ah-class LTO/NCM cell for high-energy use

Large-format prismatic cell of LTO/NCM system has been developed for EV and stationary power applications. Table 2 summarizes the specifications of the 20 Ah-class LTO/NCM cell. The LTO/NCM cell was designed for a nominal capacity of 20 Ah and

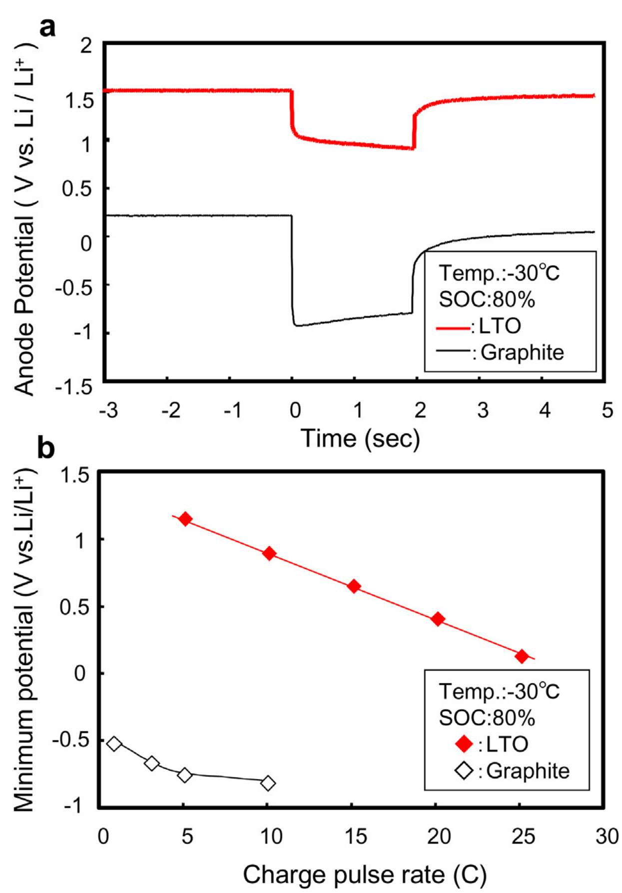

Fig. 8. Potential profiles of LTO and graphite anode for 10 C rate galvanostatic charge pulse for 2 s at −30 °C (a) and plots of minimum potential of the anodes at various rates of the charge pulse for 2 s at −30 °C (b).

Table 2
Specifications of 20 Ah-class LTO/NCM cell for high-energy applications.

Cell	20 Ah-class LTO/NCM cell
Case	Al metal can
Nominal capacity at 1C (Ah)	20
Nominal voltage (V)	2.3
Dimensions (mm)	115 × 103 × 22
Weight (kg)	0.51
10s output power (W kg^{-1})[a]	2200
10s input power (W kg^{-1})[a]	2400
Energy density (Wh kg^{-1})	90

[a] At 50% SOC and 25 °C.

an energy density of 90 Wh/kg. Fig. 9 shows the discharge rate capability at 25 °C. The discharge capacity at 8 C (160 A) rate was almost the same as that at 1 C rate. Such a discharge rate capability is high enough for EV to be competitive with conventional vehicles in terms of driving performance. Quick-charge performance at 8 C rate is shown in Fig. 10. The charge time and the cell temperature rise at 80% SOC were 6 min and 11 °C, which is charge time short enough to be acceptable for EV. Fig. 11 shows temperature dependence of discharge performance. The discharge capacity and the energy retention at −30 °C vs. 25 °C were 80 and 70%, respectively. The low-temperature performance is high enough to be acceptable for EV during the winter season in the cold latitudes.

Pulse-power capability for 10 and 30 s by HPPC testing is shown in Fig. 12. For the 20 Ah-class LTO/NCM, the maximum output and input power density for 10 s at 50% SOC and 25 °C were 2200 and 2400 W kg^{-1}, respectively. For a long-time pulse of 30 s, the maximum output and input power density at 50% SOC was 1600 W kg^{-1}. The pulse-power capability of LTO/NCM cell had a good balance of input and output. The output power densities at 20% SOC for 10 and 30 s were 1500 and 1000 W kg^{-1}, respectively, which are suitable for not only EV but also PHEV use.

Long-term storage characteristics of the 20 Ah-class LTO/NCM cell have been investigated for stationary power applications. Fig. 13 shows self-discharge performance of the 20 Ah-class LTO/NCM cells stored at 25 and 45 °C after charging to 100% SOC. The residual capacities after the long-term storage at 25 and 45 °C for 365 days were 88 and 73%, respectively. It was reported that a conventional lithium-ion cell using graphite anode stored at 45 °C showed the residual capacity of about 75% after 90 days [17]. The LTO/NCM cell showed a small self-discharge rate compared to that of the conventional lithium-ion battery. The recovery capacities after the long-term storage at 25 and 45 °C for 365 days were 100 and 99.3%, respectively. There was found to be almost no recovery capacity fading for the LTO/NCM cell after the long-term storage

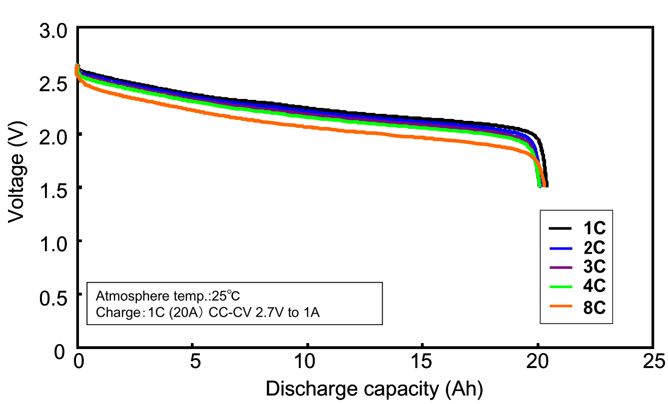

Fig. 9. Discharge curves of the LTO/NCM cell at various rates.

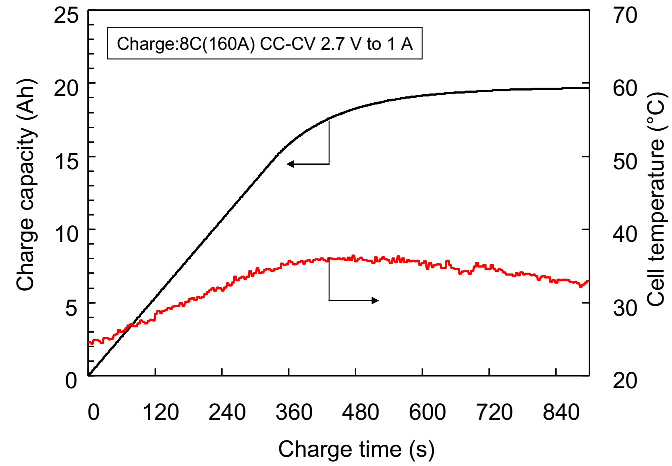

Fig. 10. Quick-charge performance of the LTO/NCM cell at 8 C rate.

even at 45 °C. We consider that such a good storage performance of the LTO/NCM cell is attributable to the fact that there is no growth of film formation on the LTO surface and only a reversible self-discharge reaction in the storage condition with open-circuit voltage. The reason for reversible self-discharge reaction in the LTO cell remains unclear and is currently being investigated. Fig. 14 shows the residual capacity fading for floating-charge storage test at 2.7 V corresponding to 100% SOC. The floating-charge storage test at a constant voltage is more severe than the storage test with the fading of open-circuit voltage by self-discharge reaction in Fig. 13. The capacity retention after the floating-charge storage at 35 and 45 °C for 250 days was 100% and 98%, respectively. The capacity retention vs. the square root of floating-charge time is plotted in Fig. 15. On the basis of the square root of time rule, the capacity retention for the floating-charge at 35 °C and 45 °C after 10 years is predicted to be 97% and 80%, respectively. Such a long storage-life is suitable for not only automotive but also stationary power use. It was reported that the capacity retention of graphite/NCM cell stored at 45 °C and 100% SOC after 2.5 years was predicted to be 70% [18]. Graphite/LMO cell for EV applications showed the capacity retention of 70% for the floating charge at 45 °C after 1 year [19]. In terms of storage-life performance at 100% SOC, the LTO/NCM cell is significantly superior to conventional lithium-ion cells of graphite/NCM system and graphite/LMO system. We consider that capacity

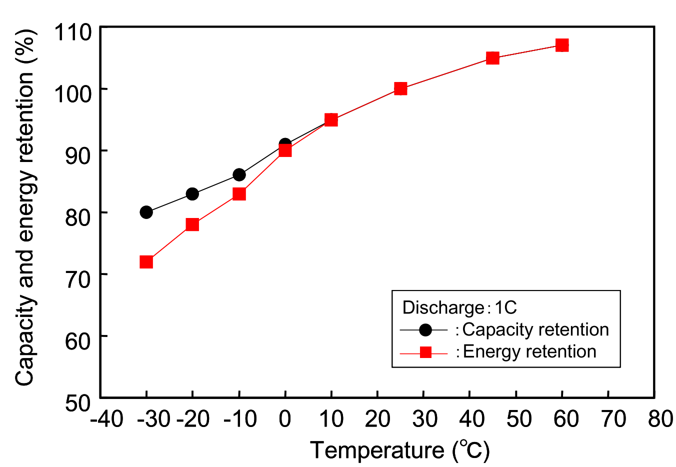

Fig. 11. Temperature dependence of discharge performance of the LTO/NCM cell at 1 C rate.

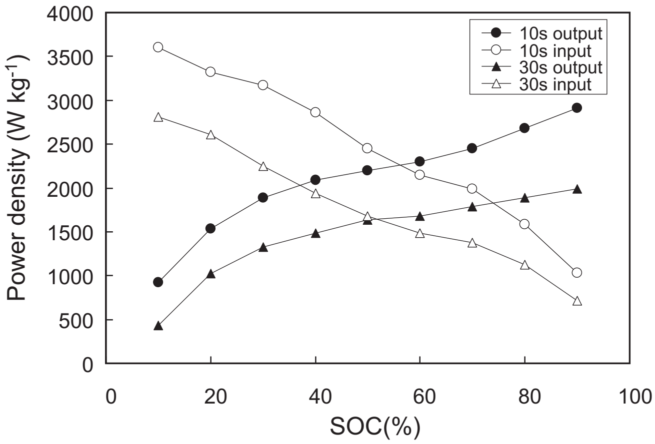

Fig. 12. Power capability of the LTO/NCM cell for 10 and 30 s at 25 °C by HPPC testing.

fading of the LTO anode during the high-temperature storage is slight compared to that of the graphite anode. The capacity fading rate of LTO cell systems is mainly dependent on the potential of cathode during the storage.

Fig. 16 shows cycle life performance and the prediction for 20 Ah-class LTO/NCM cell based on the results of the cycling test at 3 C rate between 1.5 and 2.7 V in 100% SOC width. Surface temperature of the cell during 3 C rate cycling was about 35 °C. The capacity retention after 6000 cycles was 86%. The capacity retention after 1000 cycles changes linearly with the square root of cycle number. The capacity retention after 10,000 cycles is predicted to be about 80% on the basis of the linear relationship. From the above results, the capacity fading of the LTO/NCM cells during the storage and the cycling tests is also in accordance with the square root of time rule because the capacity fading is mainly caused by side reactions and film formation at the NCM cathodes. For example, if the LTO/NCM cell stores at 35 °C and 100% SOC in the floating-charge condition, and then is fully cycled, the EOL with 80% capacity retention is predicted to be 15 years with 6000 cycles at 3 C rate for 1.5 years. The storage-life and the cycle life performance of 20 Ah-class LTO/NCM cell are long enough for EV, PHEV, and stationary power applications. It was demonstrated that the 20 Ah-class LTO/NCM cells have the excellent balance of high-power,

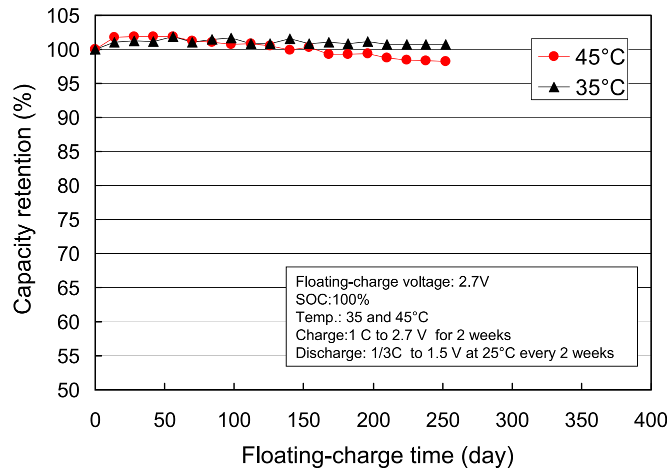

Fig. 14. Change in capacity retention as a function of floating-charge time for the LTO/NCM cells by floating-charge storage test at constant voltage of 2.7 V corresponding to 100% SOC.

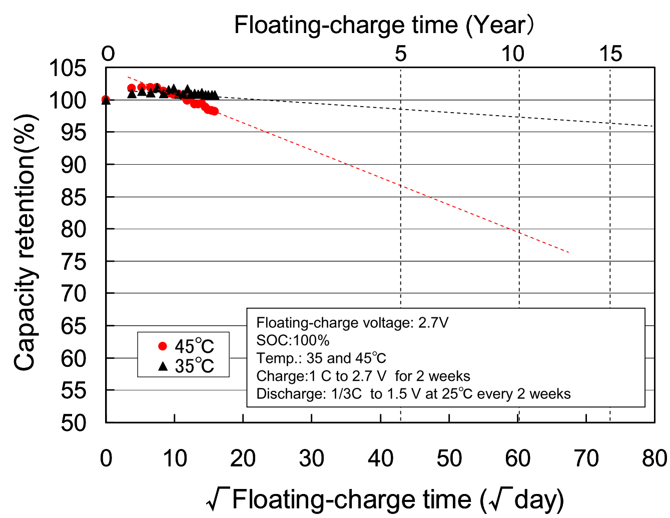

Fig. 15. Capacity retention as a function of √floating-charge time for the LTO/NCM cells at 35 and 45 °C.

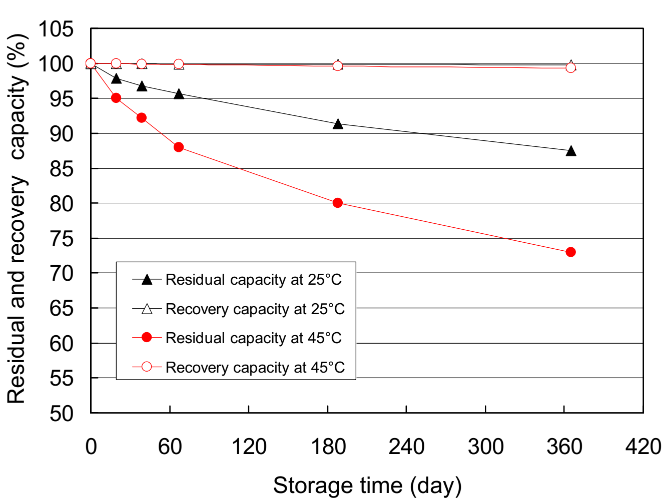

Fig. 13. Self-discharge performance and recovery capacity of the LTO/NCM cells after charging up to 2.7 V at 25 and 45 °C.

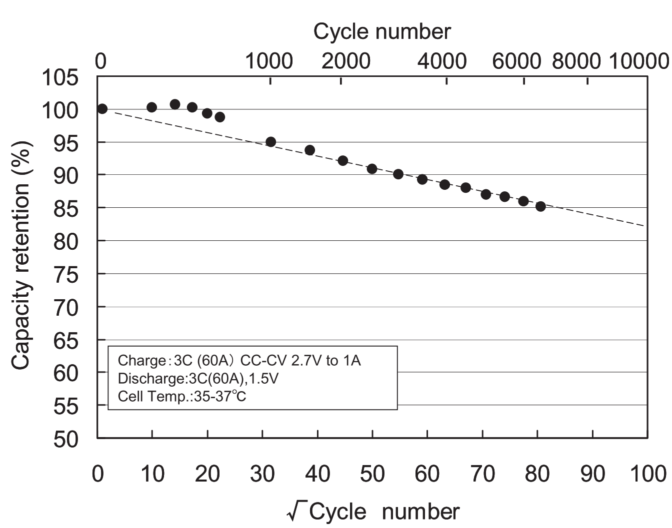

Fig. 16. Cycle life performance of the LTO/NCM cell for charge–discharge cycling at 3C rate between 1.5 and 2.7 V.

high-energy, low-temperature, and long-life performance for EV, PHEV, and stationary power applications.

4. Conclusions

Cell performance and electrochemical characteristics of LTO/LMO system and LTO/NCM system were investigated for automotive and stationary applications. It was demonstrated that the 3 Ah-class LTO/LMO cell with 64 Wh kg^{-1} for high-power applications has outstandingly high output power density of 3600 W kg^{-1} and high capacity retention of 95% after 30,000 cycles during full charge–discharge cycling test at 10 C rate. In accordance with the square root of cycle number and time rule in the full cycling test, the cycle number and cycle time at the capacity fading of 10% are predicted to be 200,000 cycles and 15 years, respectively. The capacity retention after 15 years in the calendar life test at 80% SOC and 35 °C is predicted to be 93% by extrapolating the capacity fading in accordance with the square root of time rule. The capacity fading for the LTO/LMO cells is mainly dependent on not the stress of cycling but the calendar life. Such a high-power and long-life performance of LTO/LMO cell is suitable for not only HEV but also ISS applications. The 20 Ah-class LTO/NCM cell was designed for the energy density of 90 Wh kg^{-1} and the output power density of 2200 W kg^{-1} at 50% SOC. Self-discharge tests of the LTO/NCM cell stored at 45 °C for 365 days after charging to 100% SOC showed the high recovery capacity of 99.3%, indicating a reversible self-discharge reaction due to no significant growth of film formation on the LTO anode. From the results of the floating-charge storage tests at 2.7 V, the capacity retention at 35 °C and 45 °C after 10 years is predicted to be 97% and 80%, respectively. The LTO/NCM cell is superior in terms of storage-life performance to conventional lithium-ion cells using graphite anode. The cycling test at 3C rate showed the high capacity retention of 86% after 6000 cycles, on the basis of which 80% capacity retention after 10,000 cycles is predicted. The 20 Ah-class LTO/NCM cell was demonstrated to have the excellent balance of high-power, high-energy, low-temperature, and long-life performance, which is suitable for not only EV but also PHEV and stationary power applications.

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